

Special contract SPC-94-4067

**INVESTIGATIONS OF NOVEL SURFACE  
MODIFICATION TECHNIQUES FOR WEAR  
RESISTANT Al AND Mg BASED MATERIALS**

**Item 003 REPORT ON COATING MICROSTRUCTURE AND COMPOSITION  
INVESTIGATIONS, MECHANICAL PROPERTIES EVALUATION**

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## 1. Definition of the problem.

Selection of a parameter for optimization of MDO process of B95 alloy to produce wear-resistant and antifriction surface layers is substantiated in this section. The analysis of the results of preliminary testing of OCC composition, physical and mechanical properties is carried out for this purpose. The aim of experiments is defined and an optimizing parameter is selected on this basis.

### 1.1. Analysis of optimizing object.

The analysis is carried out on the basis of the results of preliminary experiments.

The study of the influence of the composition of the silicate electrolyte on the kinetics of OCC growing, their composition and structure showed, that the growth rate of the oxide layer is minimum in the electrolyte with the concentration of 2-6 g/l. The coatings consisting basically of aluminium oxide, are formed at the current density of 15-20 A/dm<sup>2</sup>. Nonuniformity of the coating thickness in the central area and on the edges of samples is observed. Raising of silicate concentration up to 20 g/l causes the substantial increasing of OCC growth rate due to the contribution of the electrochemical mechanism to the coating forming. The coating uniformity and solid porosity are raised at the same time.

Study of the coatings microhardness showed that the hardest layers are formed in the electrolytes with silicate concentration of 2-4 g/l. Decreasing of OCC hardness, when formed in more concentrated solutions, is explored by the raising of silicate content in the coatings and their porosity.

Wear testing of the samples at ball-on-disk scheme showed, that the coatings produced in electrolyte, having the concentration of water glass around 2 g/l, possess the best antifriction characteristics and wear resistance. It was established, that tribological behaviour of friction pair at this scheme of loading is generally defined by the ratio of strength characteristics of the coating and counter-body. Besides the sufficiently limitation can be supposed due to the OCC deficient thickness, nonuniformity and porosity.

The results of preliminary testing of OCC allowed to make the following conclusions. To use the oxide layers at tribological applications it is necessary to have the coatings thickness of 100-200 μm, which will be enough to resist the contact loading, and high microhardness to resist the abrasive wear. Moreover it is required to form dense or fine-porous uniform layers to provide the antifriction characteristics.

Based on the results of preliminary testing it was defined, that further experiments in the direction of technical and economical optimization of technological regimes for production of OCC having maximum of thickness, microhardness and uniformity is expediently to carry on using the silicate-alkali electrolytes with concentration around of 2 g/l. This approach will allow to produce the most effectively oxide ceramic layers, having the given complex of physical, mechanical and geometrical characteristics, which provide the best tribotechnical parameters. Hence, the purpose of the investigation on this step was defined as follows: to determine the technological conditions of OCC forming on B95 alloy in dissolved silicate-alkali electrolytes, which provide the best combination of the coatings yield, their uniformity and microstrength.

### 1.2. Selection of an optimizing parameter.

The analysis of the basic physical-mechanical characteristics of OCC does not make it possible to determine some defined numeral parameter, which includes at the same time both the index of the process effectiveness and the strength and geometrical characteristics of the product. In connection with this the following generalized parameter is suggested for optimization of MDO-process [1]:

$$Y=Y_1(k_2Y_2+k_3Y_3) \quad (1)$$

Here  $Y_1$  is the optimizing parameter for effectiveness of the process;  $Y_2$  is the optimizing parameter for strength characteristics;  $Y_3$  is the optimizing parameter for geometrical characteristics of the coatings;  $k_2$  and  $k_3$  are weight shares of  $Y_2$  and  $Y_3$  accordingly. The first parameter is adopted to be the basic one, and the last two parameters are the restricting ones.

Number of mass of oxides, formed at the unite of oxidizing surface, is suggested as parameter of the process effectivity:

$$Y_1=M=m/s \text{ (mg/sm}^2\text{)}$$

Microhardness distribution through the coating thickness is suggested for estimation of the optimum of its strength characteristics:

$$Y_2=H(h) \text{ (GPa)}.$$

The estimation of the coatings uniformity is useful to make the distribution of the coating thickness through the sample length:

$$Y_3=h(x) \text{ (mkm)}.$$

To carry out the comparative estimation, the restricting parameters are suggested for the arrangement of the values in order to five-number scale, where 1 corresponds to the worst value of the parameter and 5 corresponds to the best one.

Taking into account the results of preliminary testing and based on the expert estimation, the weight shares  $k_2$  and  $k_3$  are adopted to be 0.5 and 0.2 accordingly.

### 1.3. Feasibility of the factors and experimental design

MDO technique is a multiple-factor process. It is characterised by the highly complicated interaction between most of them. The review on the first step of the work determined how the basic factors, such as current shape and density, electrolyte composition and temperature, silicate modulus, electrolysis duration etc., influence the physical and mechanical characteristics of OCC. Based on the results of preliminary experiments and data of ref. [2-5], the following factors can be selected which should be taken into account at the optimizing of MDO-process for tribotechnical purpose:

- electrolyte composition
- i) Concentration of water glass -  $C_1$ ;
- ii) Concentration of KOH -  $C_2$ ;
- Current density -  $I$ ;
- Electrolysis time -  $t$ .

Accounting familiar pair correlation between  $I$  and  $t$ , these factors are advantageous to merge by simplex:  $Q = It$ .

The factors basic levels, varying ranges and fixing accuracy are determined on the basis of the results of preliminary experiments (table 1).

Table 1

Ranges of the factors varying

Factor	Level			Fixing accuracy	Unit of measuring
	-1	0	+1		
C1	1	2	3	0.2	g/l
C2	1	2	3	0.2	g/l
Q	1000	1500	2000	100	A*min/dm <sup>2</sup>

At the same time other factors are fixed at the following levels:

- power on anode -  $P = 5.7 \pm 1.5 \text{ kVA/dm}^2$ ;
- current shape - AC ( $i_+/i_- = 1.0$ );
- water glass modulus -  $m = 2.15 \pm 0.1$ ;
- electrolyte temperature -  $T = 25 \pm 5 \text{ C}$ .

The accuracy of fixing for C1 and C2 is determined by an error of the used scales. But accurate fixing of Q factor is very difficult due to the regime with constant power used in the experiments. It is caused by the current density, changing in time, which variations in the manner are unknown beforehand (fig 1).

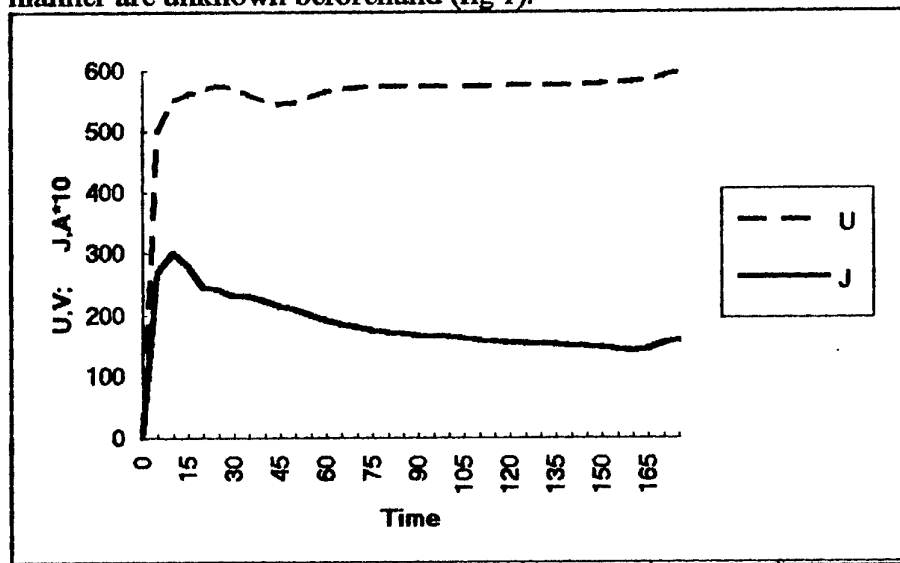


Fig. 1. Forming curves for MDO of B95 alloy in constant power regime

To keep the design of the experiment with a flutter factor to be orthogonal, the determined values of optimizing parameters were desired to approximate to the levels of -1 and +1 of factor Q according to the previously defined law [6,7].

The generalities of oxide layers anodic growth for the most of electrochemical systems with passive electrolytes are described by the equation, run out of the Faradey's ratio:

$$M = KQ. \quad (2)$$

Here K is anodizing constant.

At the same time modern information on kinetics of oxide layer growth during MDO in silicate solutions is in contradiction with (2). So, it was found in ref.[8,9], that the coating thickness is formed according to the following equation:

$$h=a(Q)^{0.88} \quad (3)$$

Linear function of the coating mass and a power, carried through the cell, was established in ref. [10] for asymmetric AC regimes. However the deficient number of experimental data, given in [10], doesn't allow to approximate them with any other function.

Generalized theoretical consideration, based on the accounting of possibility of concurrent oxide forming booth in discharge channels and surface, free of sparks, was made in [11]. The following equation was obtained to describe the coating at MDO-process:

$$M=(a_1I^{b_1}+a_2I^{b_2})t, \quad (4)$$

where  $a_1, a_2$  are proportional coefficients;  $b_1, b_2$  are indexes of a power, which depend on a function of a current, carried through the surface. It was estimated in [11], that  $a_1=0.89$  and  $a_2=1.21$ .

Obviously the general function  $M=f(I)$  can be approximated by exponential equation with the index of a power to be  $0.89 \leq a \leq 1.21$ . Thus, both equation (2) and (3) are possible.

Taking into account the reported above, the regression analysis of type of function  $M=f(Q)$  was decided to carry out. Final decision on function of extrapolation of factor  $Q$  onto +1 and -1 levels will be adopted as the result of the analysis.

## 2. Design of an experiment and a model selection.

Designing matrix for full factor experiment  $2^3$  is presented in table 2.

The orthogonal design makes it possible to determine 8 unknown coefficients of polynomial regression equation for every optimizing parameter, accounting pair and triple interactions of the factors.

Table 2.

Full factor experiment  $2^3$  design for the case of three-parameter optimization.

№ of experiment	C1	C2	Q	M	H(h)	h(x)
1	-	-	+	y11	y21	y31
2	+	-	-	y12	y22	y32
3	-	+	-	y13	y23	y33
4	+	+	+	y14	y24	y34
5	-	-	-	y15	y25	y35
6	+	-	+	y16	y26	y36
7	-	+	+	y17	y27	y37
8	+	+	-	y18	y28	y38

However, the designing of polynomial regressive equation is possible only for factor M. It is highly difficult to find a numerical variable which allowed to describe easily the behaviour of factors H(h) and h(x) at this step. The distribution of the parameters may be estimated with the help of functions of desirabilities [1].

## 3. Experimental details.

Some additions are made at this step for the procedure of the coating production and characterization, which was in detail discussed in the Report on Part II. Mass of the coatings is determined as the difference between the weight of samples before and after MDO treatment, using analytical scales of VLA-200-M model with instrumental error of 1.0 mg. Coating microhardness was measured under the loading of 0.98N to raise the accuracy of distribution  $H(h)$  definition. Error of measuring of  $H$  was  $\pm 2.5$  GPa in the range of 15-25 GPa. The coating thickness distribution through the sample length was determined by measuring the thickness on the samples cross sections with a step of  $80 \pm 10 \mu\text{m}$ , using MMR-4 microscope with  $\times 630$  magnitude when accuracy of measuring was  $\pm 5 \mu\text{m}$ .

#### 4. Results and discussion.

##### 4.1. Optimum effectiveness of OCC forming.

The estimation of effectiveness of OCC forming is carried out with the aid of interpolating model for difference of the coating mass in the discussed range of the factors. Let us carry out the regression analysis of the function of the coating mass vs. the value of electricity, carried through the cell to refine the information about kinetics of the coating growth and get the generalized knowledge of the value of the response function at different levels of fixing of parameter  $Q$ .

##### 4.1.1. Regression analysis of function $M(Q)$

Typical dependence of oxide mass deposited as a result of passing of various values of electricity is given in fig. 2.

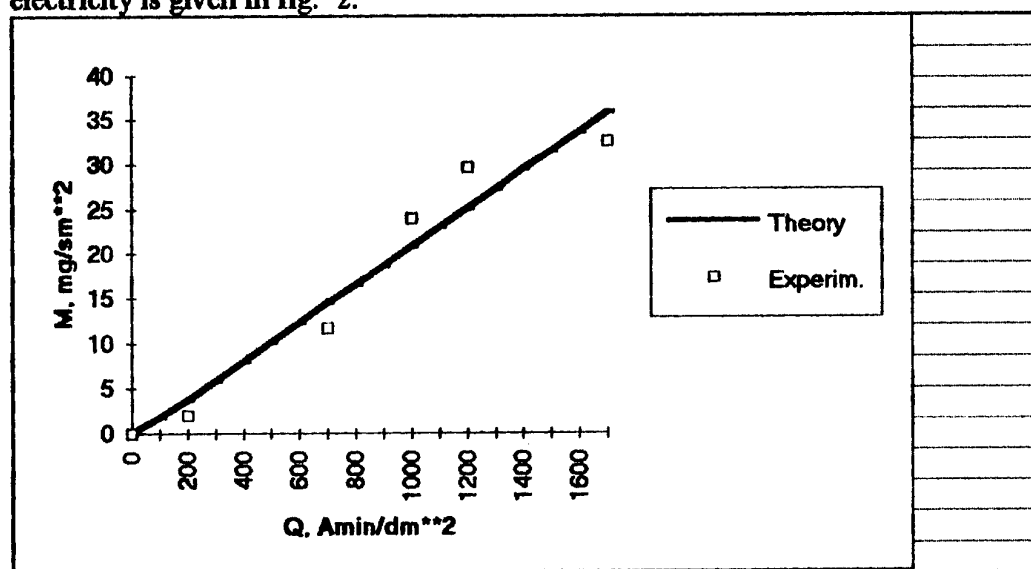


Fig. 2. Dependence of coating mass vs. value of electricity carried through the cell for MDO in 3 g/l KOH + 3 g/l  $\text{Na}_2\text{SiO}_3$  electrolyte.

Data of fig.2 shows, that the coating mass is raised in proportion to the charge carried through the sample surface. At first glance, the data obtained can be approximated by linear or exponential function having an index of a power close to 1. It corresponds to the theoretical computations discussed above. To specify the type of the function, comparative regression

analysis of the experimental data was carried out using "Stadia" software [12]. Results of the analysis are given in table 3.

Table 3.

Coefficients and adequacy parameters for function  $M=f(Q)$  in junction points of design

Function type			$Y=A + BX$							$Y=AX^B$					$F_{tab}$
N <sub>o</sub> sam	C 1	C 2	f1	f2	A	B*10 <sup>-2</sup>	Sr	Sad	F	A*10 <sup>-2</sup>	B	Sr	Sad	F	
11	-	-	1	5	1.28	1.38	173	1.68	103	9.17	0.74	117	0.003	39000	230.2
22	0	0	1	5	-0.79	1.99	1010	4.44	37.9	9.21	0.75	118	0.29	1150	230.2
33	+	+	1	5	-0.64	2.14	956	8.77	109	9.44	0.76	121	0.213	569	230.2
31	+	-	1	4	0.39	1.87	363	0.12	3080	9.62	0.75	110	0.037	2980	224.0
13	-	+	1	5	-0.86	1.42	470	6.22	75.6	7.58	0.73	113	0.18	629	230.2

Here Sr is variance of regression; Sad is variance of adequacy; f1,f2 are, corresponding to them, degrees of freedom; F is Fourier test.

Table 3 shows that the approximation by exponential function provides the best approach of data calculated to experimental ones. However the experimental accuracy at selected number of tests is deficient to declare the significance of approximation of the data by exponential function. On the contrary, F-test for the linear approximation is less then its table value almost for all points, i.e. null hypothesis can be adopted in this case.

Thus the values of response function in junction points of factor Q would be determined using linear approximation of experimental data with the coefficients corresponding to table 3.

#### 4.1.2. Results of parameter M optimizing

Design matrix of  $2^3$  experiment for the coating mass is shown in table 4. Coefficients  $b_i$  of polynomial regression equation were determined by the method of minimum squares. Tests of model for adequacy and regression coefficients for regression were carried out with the help of "Stadia" software. It is shown that variance of regression coefficients  $S\{b\} = 0.3535$ .

Table 4

Design matrix of  $2^{3*3}$  for parameter M

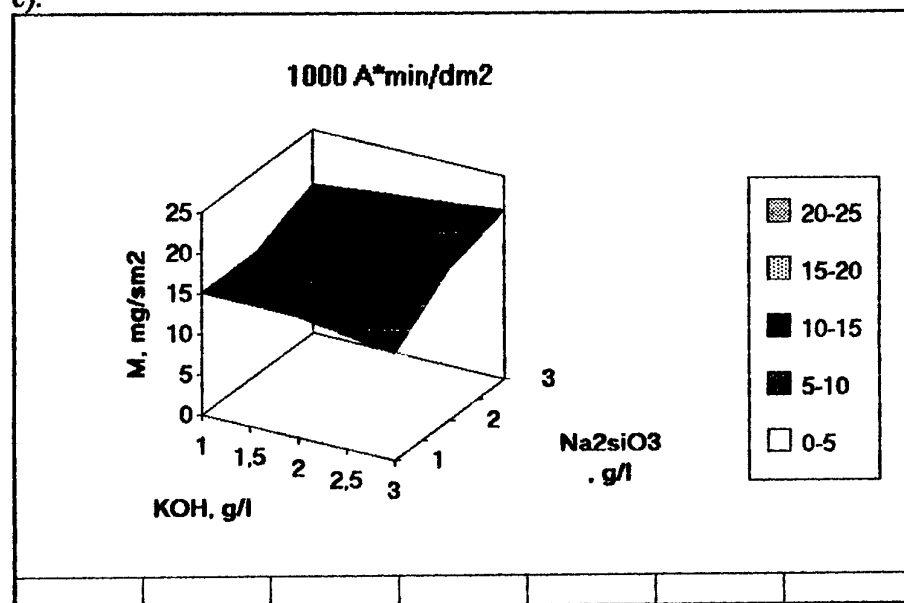
N sam	x0	x1	x2	x3	x1x2	x1x3	x2x3	x1x2x3	Y
113	+	-	-	+	+	-	-	+	28.9
311	+	+	-	-	-	-	+	+	18.3
131	+	-	+	-	-	+	-	+	13.3
333	+	+	+	+	+	+	+	+	42.2
111	+	-	-	-	+	+	+	-	15.1
313	+	+	-	+	-	+	-	-	37.5
133	+	-	+	+	-	-	+	-	27.5
331	+	+	+	-	+	-	-	-	20.8
$b_i$	25.4	4.2	0.55	8.525	1.35	1.525	0.375	0.275	

Taking into account value of Student coefficient for 5% regression level,  $t = 2,766$ , it is easily to calculate, that error of determination of regression coefficients will be  $Db = 1.125$ . So,

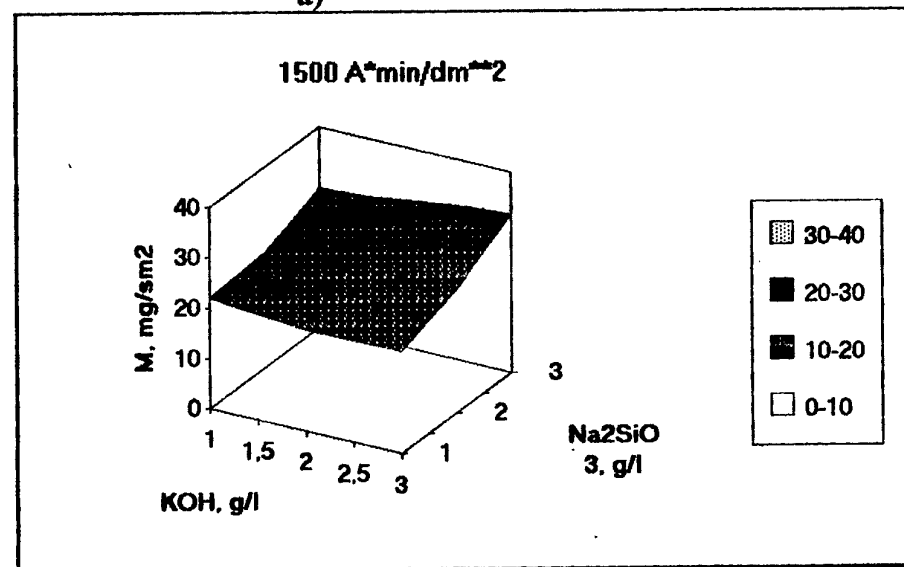
coefficients of b2, b6 and b7 are insignificant. Finally, the regression equation for the studied parameter will be the following:

$$M = 25.4 + 4.2 C1 + 8.525 Q + 1.35 C1C2 + 1.525 C1Q. (5)$$

Response surfaces of the coating mass for different levels of factor Q are shown in fig 3 (a-c).



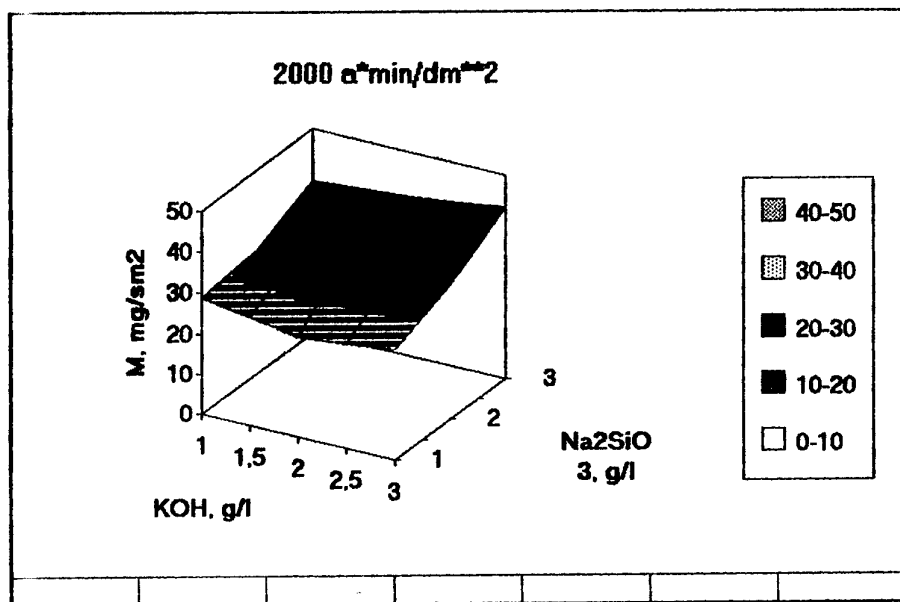
a)



b)

As follows from the analysis of fig.3 with accounting of eq.(5) the value of carried electricity is the basic factor defining mass change of the coating. Alkali concentration C1 is the second significant factor. It's remarkable, that factor of silicate concentration C2, given along, insufficiently influences the coating mass change. At the same time pair interaction of alkali and silicate C1C2 is substantial. It is the most clear on the top level of factor Q (fig.3 (c)) and confirmed by the regression of coefficient for pair interaction of factors C1 and Q in eq. (5).





c)

fig. 3. Response surfaces of the coating mass at values of  $Q = 1000$  (a),  $1500$  (b) and  $2000$  (c)  $\text{Amin/dm}^2$ .

Touching upon the interpretation of the results in terms of physics of the phenomena, the following facts should be noted. In the first place, the maximum influence of value of electricity and alkali concentration on the mass change is obviously connected with the familiar ratio (2). Moreover, the increasing of electrolyte conductivity is the main positive effect of raising of alkali concentration. It reduces total resistance of the cell and is equal to the additional setting up of the current density. On this basis, the pair interaction of factors of  $Q$  and  $C1$  can be interpreted as the additional value of electricity carried through the cell because of the current density was set up when electrolyte conductivity was raised. The fact, that increasing of water glass concentration in the studied area insufficiently influences the coating mass change, indicates low charge of silicate ions in the solution. In this consequence, their contribution, as sources of oxygen, into the coating forming isn't great in comparison with  $\text{OH}$  ions and water. OCC formed in the given range of concentrations would be considered as pure alumina films. Silicate actions are displayed only when combined with alkali. It indicates that additions of water glass generally act on oxidizing surface as passivators which balance out aggressive action of alkali on the coating. The more alkali is contained in the electrolyte, the more silicate is necessary to achieve the stable passive state of an anode. Disbalance of the contents of alkali and silicate in electrolyte may lead to decreasing of stability of passive state, on the one hand, or to reduction of electrolyte conductivity and raising of the process time - on the other hand.

Thus, the following conclusions can be made based on the results of the analysis of response surfaces and regression equation for oxide mass yield from volume of electricity, carried through the cell:

- i) The coating mass yield in the studied range of the factors is adequately described by equation (5) with 1.125 error;
- ii) Value of electricity and concentration of  $\text{KOH}$  are the basic factors, which influence the coating mass change;
- iii) Silicate acts only as inhibitor in this area and does not influence sufficiently the performance of oxide forming itself. Maximum positive silicate action is displayed when it is combined only in equal parts with alkali;
- iv) The concentration range of 2-3 g/l  $\text{KOH}$  and 2-3 g/l water glass would be considered as optimum for effective OCC forming.

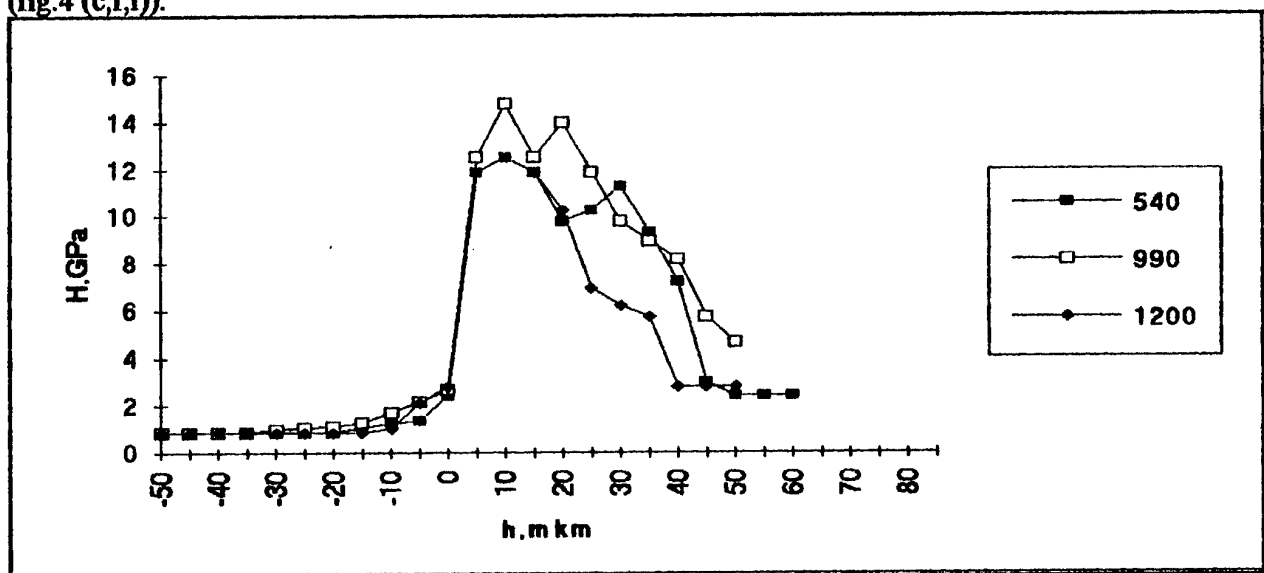
## 4.2. Analysis of restricting factors $H(h)$ and $h(x)$ action

### 4.2.1. Microhardness distribution.

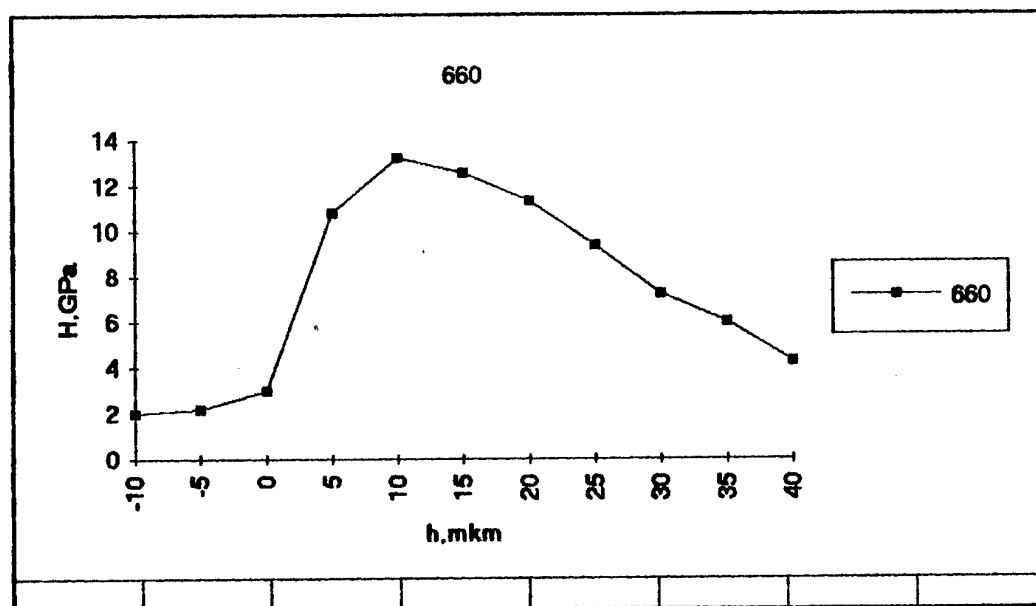
Analysis of microhardness distribution through the coating thickness was aimed to estimate strength characteristics of OCC in the studied range of factors. Both position, volume of maximum microhardness  $H_{max}$  and its gradient were taken into account in the analysis.

The curves of microhardness distribution at junction points of design matrix are given in fig.4 for various  $Q$ . Maximum value of coating microhardness is observed in the points, where factor  $C2$  is fixed at a low level. When silicate concentration increases, the microhardness drops for the coatings produced at the same  $Q$ .

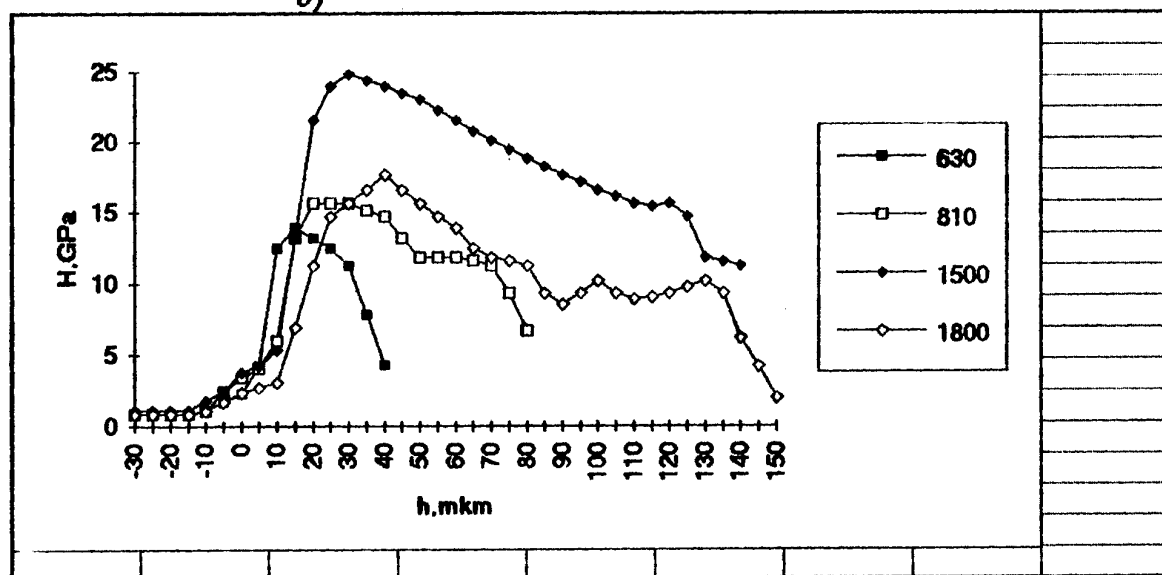
General tendency to increase the maximum microhardness is shown with raising the value of electricity upto  $1500 \text{ Amin/dm}^2$ . When more  $Q$  is carried through the cell, the rate of  $H_{max}$  is decelerated. This trend is more prominent, when factor  $Q$  is fixed at top level. Even decreasing of  $H_{max}$  is observed here after more than  $1500 \text{ Amin/dm}^2$  was carried through the cell (fig.4 (c,f,i)).



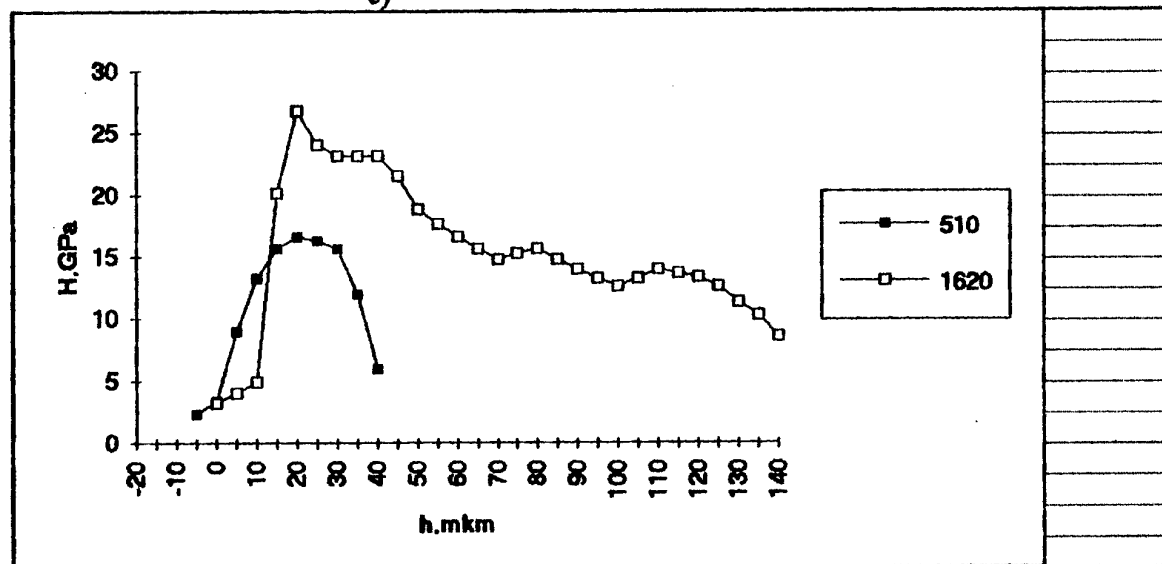
a)



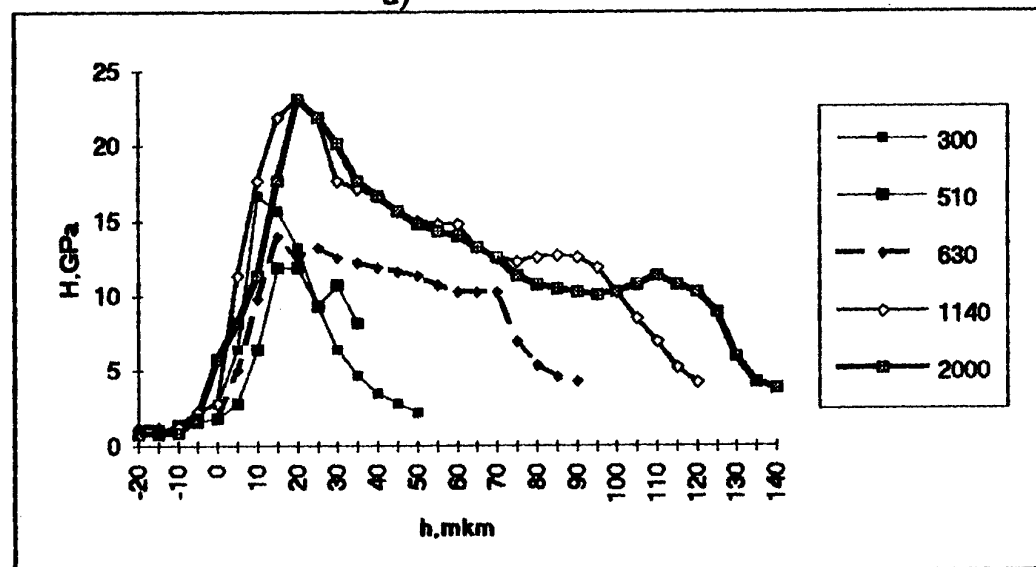
b)



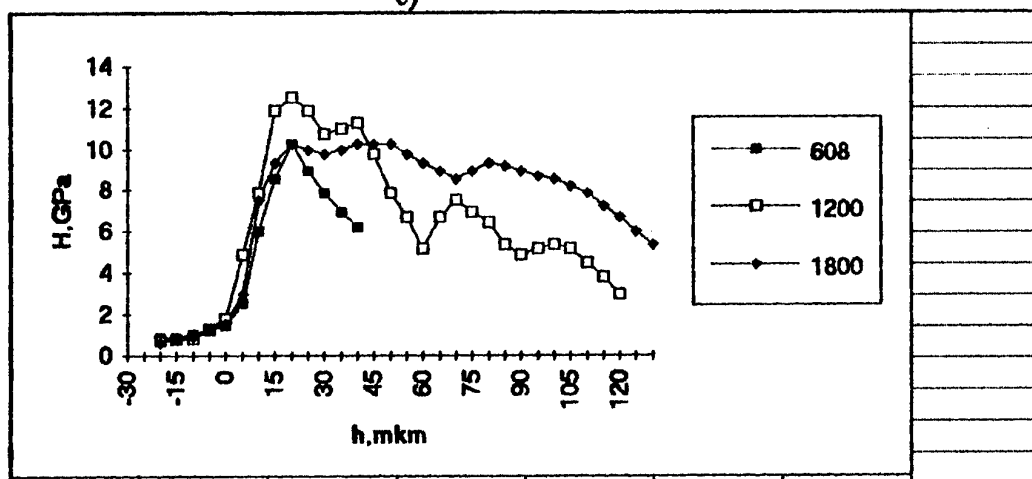
c)



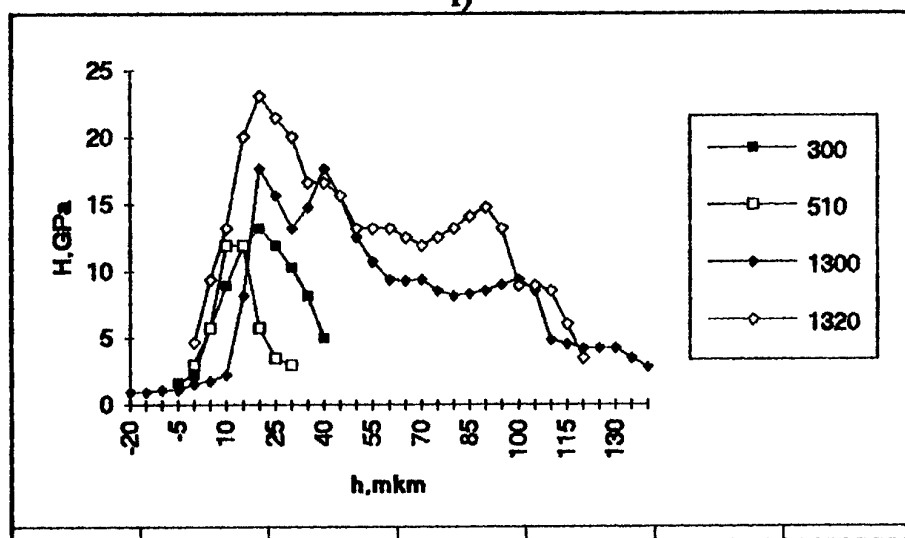
d)



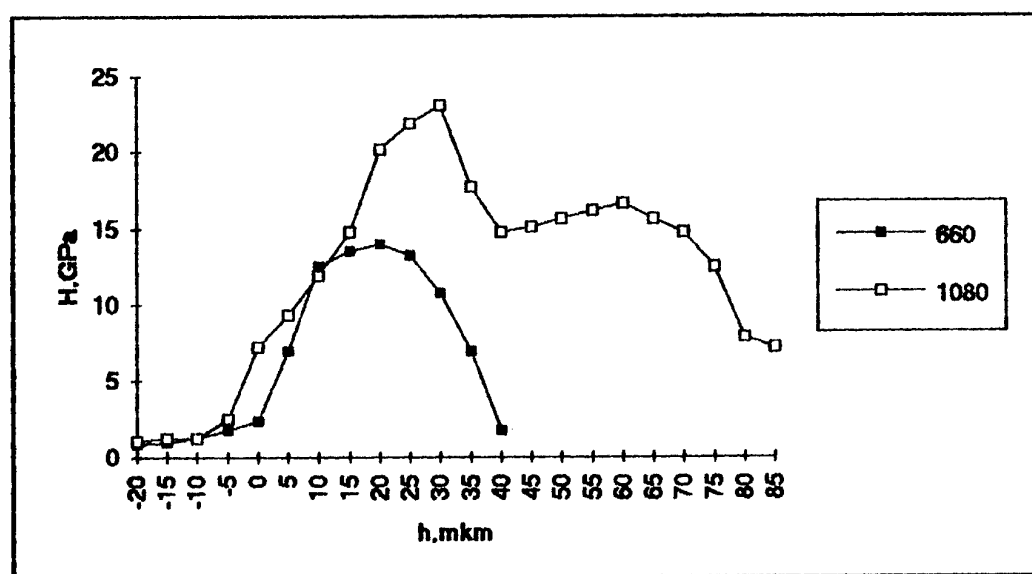
c)



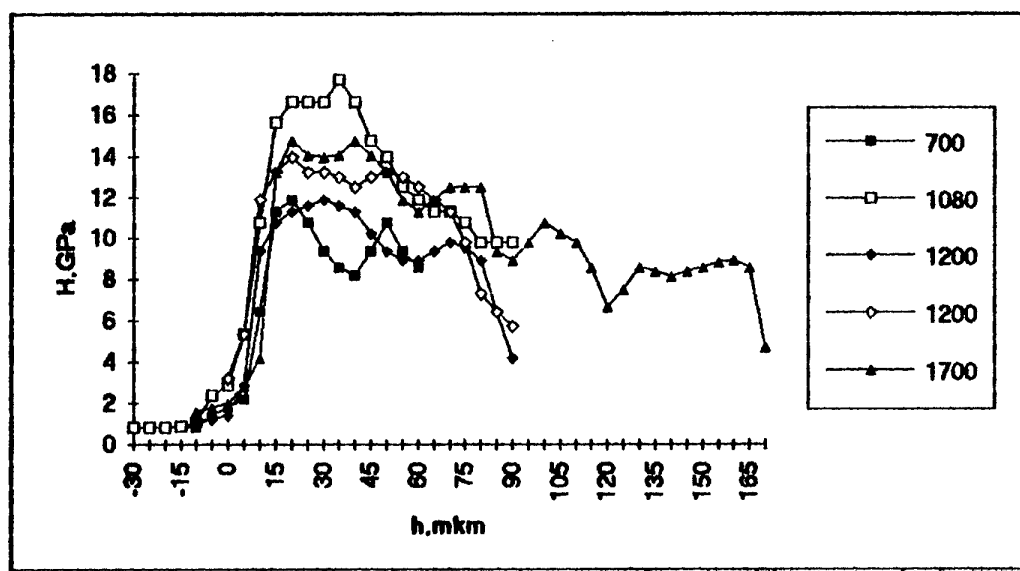
d)



e)



h)



i)

fig. 4. Microhardness distribution through the coating thickness for MDO in silicate-alkali electrolytes with concentrations of  $\text{KOH}(\text{g/l}) + \text{Na}_2\text{SiO}_3(\text{g/l})$ : a)- 1+1; b)- 1+2; c)- 1+3; d)- 2+1; e)- 2+2; f)- 2+3; g)- 3+1; h)- 3+2; i)- 3+3. Values of carried electricity are indicated in chart legends.

Position of  $H_{\max}$  depends on the studied factors too. The following regularities are the general tendency here: the coordinate of  $H_{\max}$  is maximum and accounts for  $40 \pm 5 \text{ mkm}$  from the metal-coating interface, when all factors are fixed at top levels (fig.4(i)). This value accounts for  $25 \pm 5 \mu\text{m}$  at the center point of design (fig.4(e)) and  $20 \pm 5 \mu\text{m}$  for low level of all factors (fig.4(a)). Microhardness gradient shows the trend for decreasing with raising of  $Q$  for all samples.

Maximum grad  $H$  are observed for the coatings, produced at low level for all factors (fig.4(a)). It should be noted, that increasing of factors level, of  $C_2$  and  $Q$  especially, leads to irregular changing of grad  $H$  through the layer thickness (fig.4(f,h,i)). The coatings, produced in this area, are characterized by jerky change of microhardness value, which indicates variations of phase composition, which causes the fluctuations of physical-mechanical characteristics. Some raising of porosity of the coatings, produced in this region, is possible too, which can influence the oscillation of  $H(h)$  function.

Analysis of desirability of achieving of a given microhardness distribution, based of the coatings application in friction pairs, shows, that the distribution with the combination of maximum values of  $H_{\max}$ , its coordinate and minimum gradient of microhardness through the coating thickness, should be accounted as optimum.

As we can see from data of fig.4, no coatings possess such combination of distribution characteristics. Attempts to produce high value of maximum microhardness in electrolytes, having high concentration, failed, but the position of maximum and gradient of microhardness were the best. The coating microhardness is raised, when the concentration of electrolyte components is reduced, but the position of  $H_{\max}$  is shifted closely to the substrate and microhardness gradient through the coating thickness is raised.

Therefore, it is expedient to use generalizing function of desirability to estimate microhardness distribution:

$$D = \left[ \prod_n d_i \right]^{1/n}$$

Here  $d_i$  is partial function of desirability for  $H_{max}$ ;  $x(H_{max})$  and  $gradH$ , defined from equation:

$$d_i = \exp(-\exp(-g_i)),$$

where  $g_i$  is order of desirability for  $i$ -factor.

Taking into account the influence of varied factors on the characteristics of  $H(h)$  distribution, the matrix of response function for parameter  $H(h)$  can be determined as follows Table 5.

Table 5.

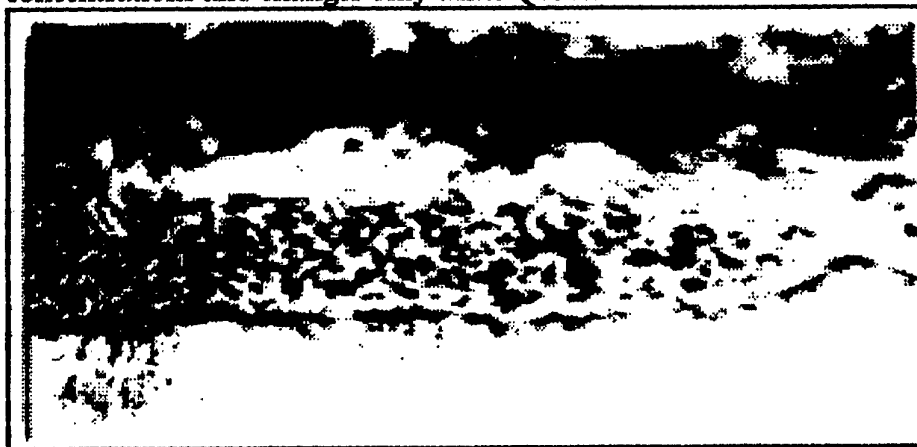
Response matrix for microhardness distribution accounted for function of desirability.

Q = 1000 Amin/dm <sup>2</sup>				Q = 1500 Amin/dm <sup>2</sup>				Q = 2000 Amin/dm <sup>2</sup>			
C1\C2	1	2	3	C1\C2	1	2	3	C1\C2	1	2	3
1	0.36	0.37	0.50	1	0.28	0.37	0.68	1	0.36	0.37	0.50
2	0.39	0.57	0.43	2	0.73	0.75	0.43	2	0.73	0.77	0.49
3	0.52	0.66	0.61	3	0.62	0.66	0.69	3	0.62	0.66	0.48

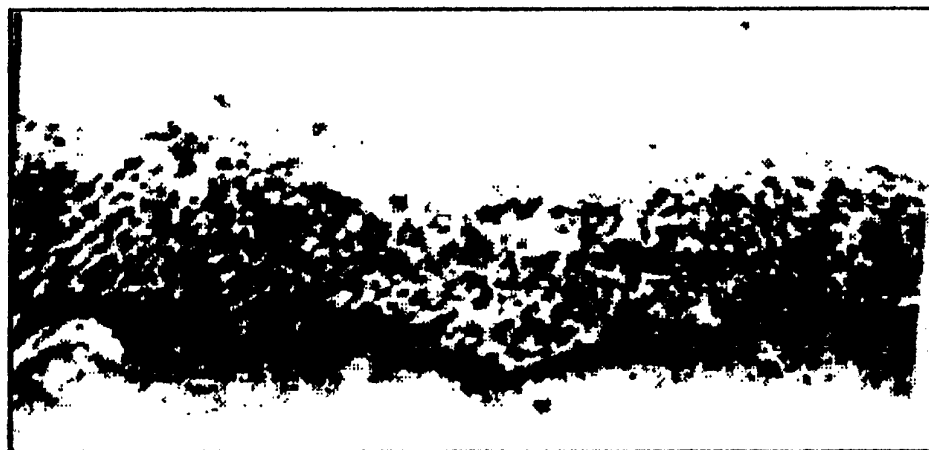
As follows from the data of Table 5, the coatings, produced at conditions, corresponding to the center and top points of C1 and Q factors, possess the most desirable microhardness distribution. While the strength characteristics are optimum for the centre point of design as well as for its projection on the top level of Q factor.

#### 5.2.2. Distribution of the coating thickness through the sample length.

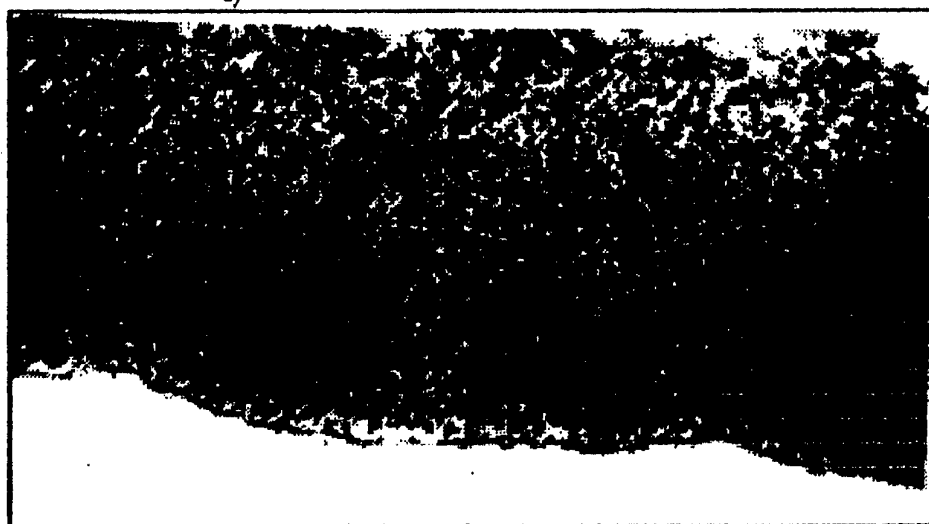
Microhardness of OCC cross sections, produced at various Q, are presented in fig.5. It's clear that the coating structure consists of dispersed dark incorporation uniformly distributed in light matrix. Basically, the coatings structure is of the same type for all studied range of electrolyte concentrations and changes only when Q is raised.



a)



b)



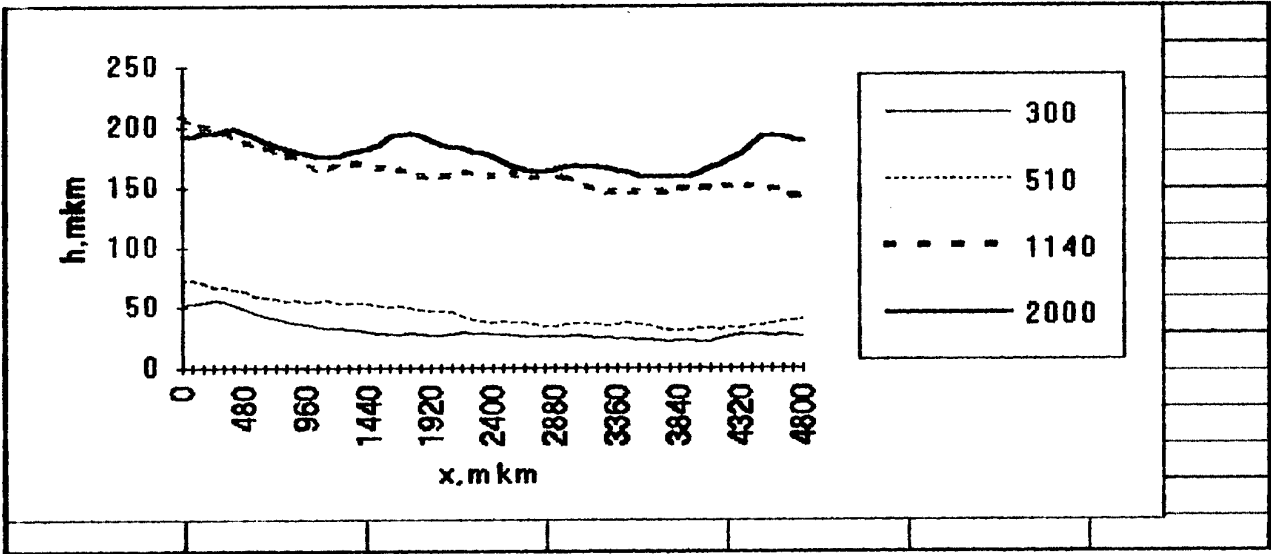
c)

fig. 5. Micrographs of cross sections of oxide ceramic coatings produced in 2 g/l KOH + 2 g/l  $\text{Na}_2\text{SiO}_3$  electrolyte at  $Q = 300$  (a); 630 (b) and 2000 (c)  $\text{Amin/dm}^2$ . Mag.  $\times 450 \times 4$ .

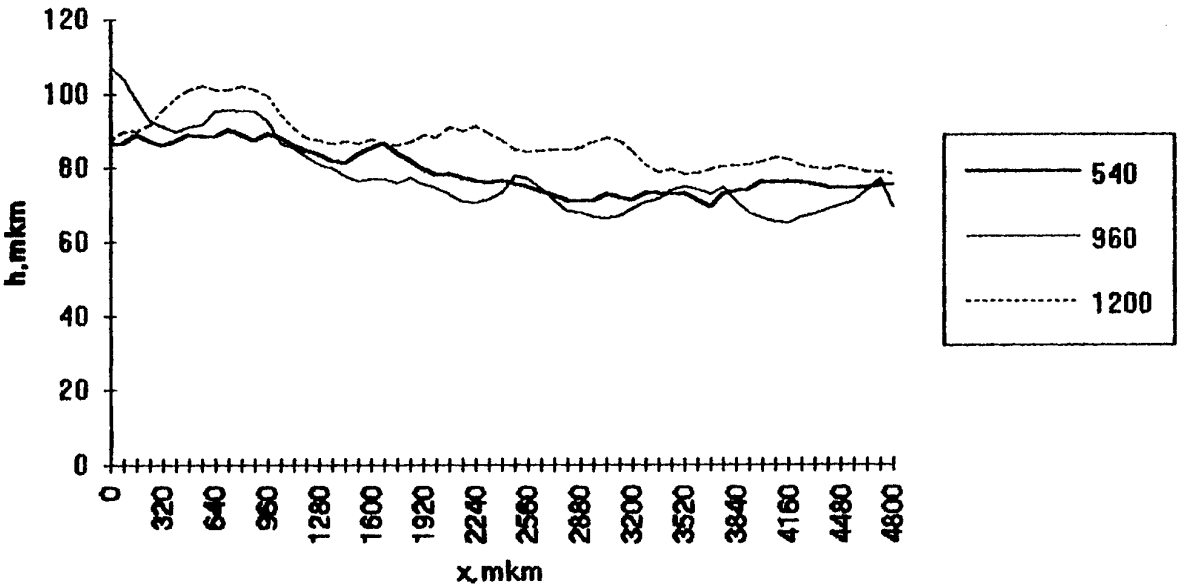
Three basic zones - external porous, central and transient to metal ones, are discerned in the cross sections of coatings produced at  $Q < 1500 \text{ Amin/dm}^2$  (fig 5 (c)). The coating thickness variance is defined mainly by changes in external porous and transient layers thickness. The structure of external layer is formed in the result of hydrodynamic action of discharges in the coating-electrolyte interface. Transient layer is absent at low  $Q$  (fig. 5 (a)). It is formed on the top level of this factor because of the combined action of oxygen diffusion into a metal and electric discharges, which melt out depressions in metal surface. Three levels of the coating thickness nonuniformity can be discerned. Microroughness having a period of less than  $5 \mu\text{m}$  sets up the first order of nonuniformities. They are located in the range of external layer and represent itself as the roughness formed in a discharge zone as a result of condensation of discharge channel substance contacting with an electrolyte. Nonuniformities of the second order have a period of  $10\text{-}50 \mu\text{m}$ . They represent the surface profile formed as a result of multiply action of discharges on the surface. Nonuniformities of the third order are the macro-inhomogeneities of the coating thickness through the sample length. Their period lies in the highly wide range from  $0.05$  to  $2\text{-}3 \text{ mm}$ . They are formed as a result of nonuniform electric field at various areas of the sample. Obviously, that macro-inhomogeneities have a maximum effect on the coatings friction

characteristics. That's why MDO-technique optimizing on the restricting parameter  $h(x)$  is expediently to carry out by the analysis of distribution of macro-inhomogeneities through the coating length.

Profiles of coatings produced at different design points are presented in fig. 6.

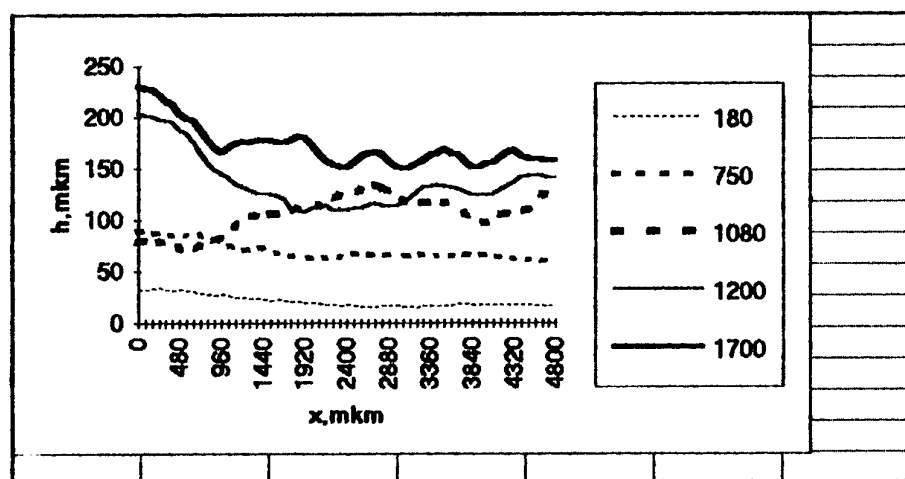


a)



b)



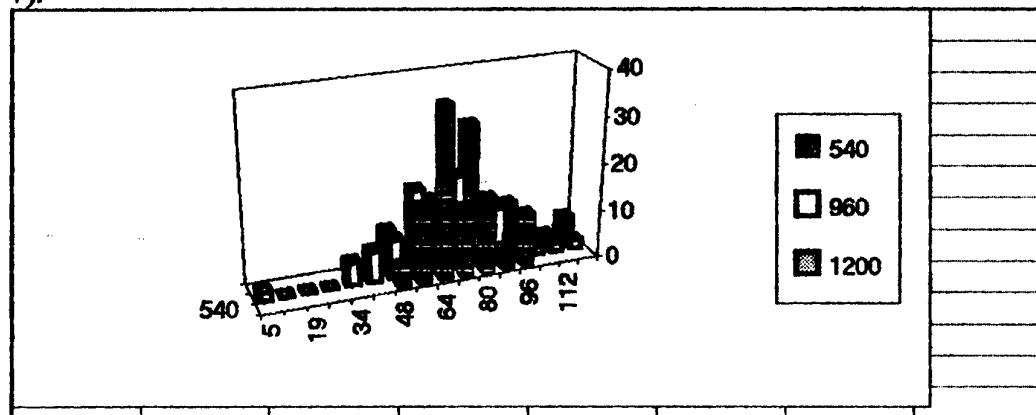


c)

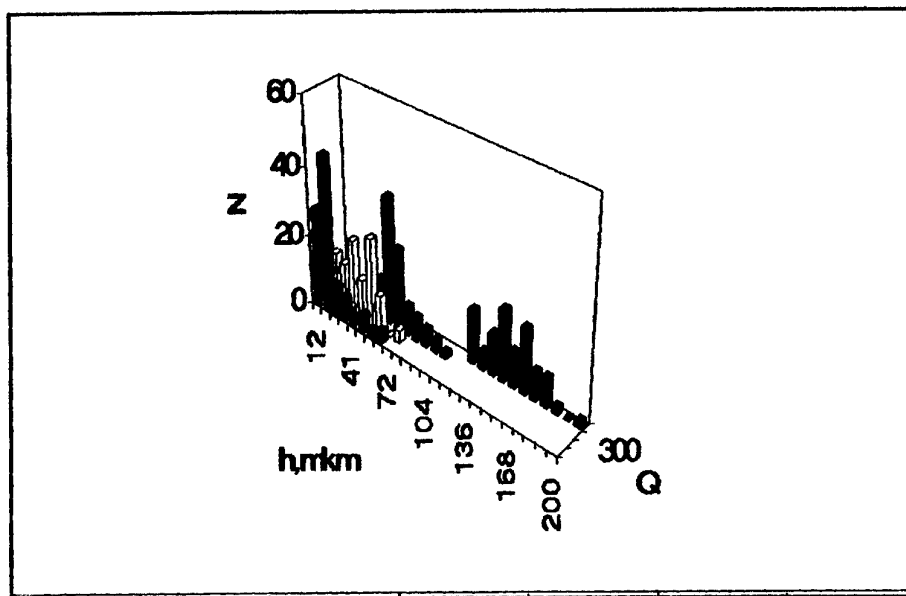
fig. 6. Coatings thickness profiles through sample length, when treated in silicate-alkali solutions with concentrations of KOH (g/l) + Na<sub>2</sub>SiO<sub>3</sub> (g/l): a)- 1+1; b)- 2+2; c)- 3+3. Values of carried electricity are indicated in chart legends.

The curves were plotted using exponent smoothing over experimental dots. In every case the origin point corresponds to edge of sample. It's clear that raising of coating thickness on the length of 0.5-1.0 mm from the sample edge is a general trend for all samples. At low  $Q$  the coatings are uniform enough and increasing of nonuniformity of the coating thickness is observed at  $Q = 600-1300 \text{ Amin/dm}^2$ . This effect is displayed more plainly at top and low levels of factors of C1 and C2 (fig. 6 (a,c)). At center point it is less perceptible (fig. 6 (b)). At further raising of  $Q$  the nonuniformity is slightly reduced. Visual inspection of the coatings produced in this range allowed to find the presence of uniformly distributed fine light islets which are formed as a result of multiply action of discharges in the same point of the sample. It leads to the local increasing of the coating thickness, which is well seen on the profiles of fig.6 (c). Spacings between islets are reduced when the coating thickness is raised. The full obliteration of the coating with a new light substance takes place at the center and top levels for all factors. At low levels the coating nonuniformity is kept.

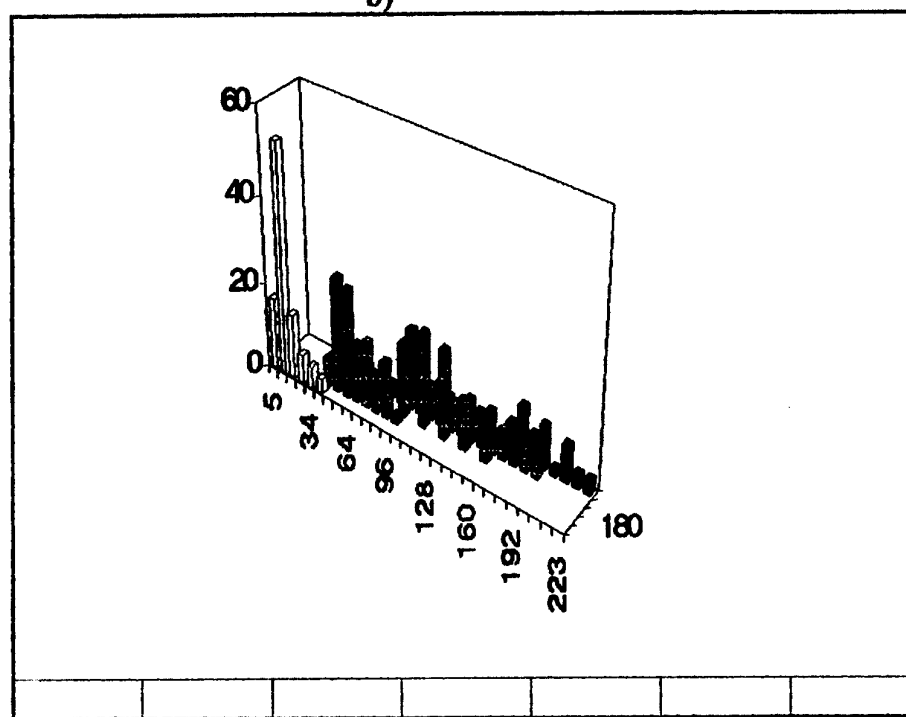
The test for normal distribution was carried out and bar charts of the coating thickness at various design points were plotted to estimate the degree of the coating nonuniformity (fig 7).



a)



b)



c)

fig. 7. Bar graphs of distribution of thickness for the coatings produced in silicate-alkali solutions with concentrations of KOH (g/l) + Na<sub>2</sub>SiO<sub>3</sub> (g/l): a)- 1+1; b)- 2+2; c)- 3+3. Values of carried electricity are indicated in chart legends.

Analysis of data of fig. 7 shows that the distribution follows the normal law at low values of Q. Maximum corresponding to derivation from the mean value both in smaller and larger sides are arised when Q is increased upto 1000 Amin/dm<sup>2</sup>. It means that the areas in the coating surface are formed in concurrent with the islet outgrowths, where oxide layer does not change and more decreasing of its thickness is observed (fig 7.(a)). There is the evidence of loss of stable passive state of the surface in those areas. The presence of the areas negatively influences both the wear-resistance and antifriction characteristics of the coatings. It may be the reason for oscillations of friction coefficient observed at preliminary wear testing.

Taking into account discussed features of forming of OCC profile the parameter of  $h(x)$  was estimated by the following characteristics: coating mean thickness  $h$ , shape of distribution histogram and the value of maximum thickness at the edge of sample.

The procedure of determination of generalized function of desirability for distribution of coating thickness through the length was in correspondence with the one for  $H(h)$  parameter. Accounting the function of desirability, the response matrix for parameter  $h(x)$  was calculated for various levels of factor  $Q$  (table 6).

Table 6

Response matrix for OCC thickness distribution through sample length accounting the function of desirability

$Q = 1000 \text{ Amin/dm}^2$				$Q = 1500 \text{ Amin/dm}^2$				$Q = 2000 \text{ Amin/dm}^2$			
C1\C2	1	2	3	C1\C2	1	2	3	C1\C2	1	2	3
1	0.22	0.37	0.45	1	0.38	0.37	0.72	1	0.38	0.67	0.75
2	0.18	0.20	0.40	2	0.27	0.77	0.74	2	0.73	0.82	0.76
3	0.33	0.48	0.45	3	0.68	0.72	0.75	3	0.62	0.77	0.79

Thus, the analysis of coating thickness distribution through the sample length showed, that OCC having optimum  $h(x)$  are formed in the range of center and top level of factors  $C1$  and  $C2$ , when factor  $Q$  is maintained at top level.

### 5.3. Total optimization of MDO-process

Estimation of summarized influence of restricting parameters of optimization was carried out taking into account eq.(1). Table 5 presents final response matrix of restricting parameters  $H(h)$  and  $h(x)$  accounting their weight shares for top level of factor  $Q$ .

Table 7

Response matrix for parameters of  $H(h)$  and  $h(x)$  at top level of factor  $Q = 2000 \text{ Amin/dm}^2$

C1\C2	1	2	3
1	2.52	3.19	4.00
2	5.11	5.49	3.97
3	4.34	4.84	3.93

Let us find generalized response function of parameter  $Y$  of technical-economical optimization for MDO-process substituting the data of table 7 into eq. (1).

Results of calculation of response function are given in fig. 8. As we can see from the data of fig. 8 the optimum region of  $Y$  corresponds to the central level for factors  $C1$  and  $C2$  and to top one for  $Q$ .



pravdopodobiya. Moscow, "Nauka", 1964.

7. V.V.Fedorov. Teoriya optimalnogo experimenta. Moscow, "Nauka", 1971.

8. Gordienko P.C., Rudnev V.C. O kinetike obrazovaniya MDO-pokrytii na splavakh aluminiya. // Zashchita metallov.-1990, t.26, 3,-P.467-470.

9. Rudnev V.S., Gordienko P.C. Zavisimost tolshchiny pokrytiya ot potenciala MDO // Zashchita metallov.1993, t.29, 2. -p.304-307.

10. Bulychev S.I., Fedorov V.A., Danilevskij V.P. Kinetika formirovaniya pokrytii v protsesse mikrodugovogo oksidirovaniya. // Fizika i chimiya obrabotki materialov. 1993, N6, p.53-59.

11. Yerokhin A.L. Fiziko-chimicheskie protsessy pri plazmenno-elektroliticheskoi obrabotke splavov aluminiya v silikatnykh elektrolitakh. //Thesises ... cand. of science degree. Tula, 1995. 240p.

12. Analiz dannykh na personalnom komputere. // Edited by E.N.Figurnov. - Moscow, "Nauka", 1989, 254p.

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